

11/01 '06 MER 15:18 FAX 418 545 4137

PYROTEK INC. CHICOUTIMI

002

Docket No.: 4715-006

PATENT

## IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of

Sylvain P. TREMBLAY

U.S. Appl. Serial No. 10/722,447

Filed: November 28, 2003

:  
:  
:  
:  
:  
Group Art Unit: 1742  
:  
Examiner: Scott R. Kastler

For: FREE FLOWING DRY BACK-UP INSULATING MATERIAL

Commissioner For Patents  
P.O. Box 1450  
Alexandria, VA 22313-1450DECLARATION UNDER 37 CFR §1.132

Now comes Sylvain P. Tremblay, who deposes and states that:

- 1) I am the inventor of the present invention;
- 2) I have an engineering degree in metallurgy, and a master's degree in ceramics;
- 3) I worked for Alcan International for 10 years, where I conducted research and development (R&D) in material recycling, high tech ceramics and refractory research. For the past 10 years, I have worked for Pyrotek, Inc., where I have been involved with product development related to the aluminum industry. The principal projects therefor have been 1) refractory castable development, 2) development of aluminum refining agents, and 3) development of aluminum distribution in the ingot head.

4) Attached to this Declaration and incorporated herein is a bibliography of technical publications co-authored by me since 1984 to the present.

5) I have read the Official Action of December 6, 2005, and make the following comments and conclusions below regarding the differences between the presently claimed free flowing dry back-up insulating material and the products of Doza et al., U.S.

6,458,732.

During an Interview with Examiner Kastler on October 14, 2005, samples of the presently claimed insulating back-up material ("PYROFLOW") and of Doza et al. ("DRI-LITE 65A"), as manufactured by the Assignee, Allied Mineral, were displayed for inspection along with corresponding product specification sheets as well as a product specification sheet for "DRI-LITE 79 AC."

Applicant's U.S. representative has retained these samples, and they are, thus, available if the Examiner deems further inspection to be warranted.

Although back-up insulating materials are known in the aluminum industry, such as WOOLITE®, such materials entail the use of water in their preparation. Hence, any residual water must be removed completely as the back-up insulating material comes into contact with molten aluminum. This has proved to be

problematic. See page 1, lines 11-30 of the present specification.

Other conventional back-up insulating materials, such as DRI-LITE®, are not free-flowing and require the use of a vibrator for installation. See page 2, lines 1-5 of the present specification.

The subject matter of present claims 1-10 avoids the above disadvantages by providing a back-up insulating material which is, at once: 1) water-free, 2) free-flowing, 3) of low density, 4) of low thermal conductivity and 5) free of organics, particularly organic binders.

In particular, present claim 1 recites a free-flowing dry back-up insulating material having a thermal conductivity ranging between about 0.8 and about 1.8 BTU·in/ft<sup>2</sup>·h·°F, and a setting temperature lower than 400°F, wherein the material consists essentially of:

a) from 67 to 96% by weight of fly-ash containing cenospheres,

b) from 2 to 15% by weight of a heat-sensitive binder selected from the group consisting of boric acid and anhydrous boron oxide,

c) from 2 to 7% by weight of a non-wetting agent selected from the group consisting of calcium fluoride, magnesium fluoride and barium sulphate,

d) from 0 to 10% by weight of a heat expandable material selected from the group consisting of vermiculite and graphite; and

e) from 0 to 1% by weight of a dust suppressant.

In contrast, Doza et al. merely describe a back-up insulating material which contains: 1) a filler light weight material, and 2) a matrix material. A heat-activating bonding agent may be used, but is clearly optional.

Notably, this reference describes the use of many different filler light weight materials, which may be selected from:

...perlite, vermiculite, expanded shale, expanded fireclay, expanded alumina silica hollow spheres, bubble alumina, sintered porous alumina, alumina spinel insulating aggregate, expanded mullite, cordierite, and anorthite...See the Abstract.

Further, Example 1 at columns 12-13 describes the use of perlite as the filler light weight material, while Example 2 at columns 13-14 describes the use of alumina/spinel insulating aggregate as this material. Importantly, this reference provides neither a description nor a suggestion to use fly-ash containing cenospheres in a back-up insulating composition.

Attached hereto are three Product Bulletins, one for DRI-LITE® 79AC and another for DRI-LITE® 65A, of Allied Mineral Products, the assignee of Doza et al.. A third product sheet for "PYROFLOW" of the present invention as claimed is also attached. Several points are worthy of note.

First, both the 65A and 79AC product sheets describe that these "aluminum-silicate based insulating dry-vibratable" products contain in excess of 80% of  $\text{Al}_2\text{O}_3$  (alumina) and  $\text{SiO}_2$  (silica). Thus, the total amounts of alumina and silica used in these products are comparable to the amount of fly-ash containing cenospheres used in present claim 1, and as exemplified by the Example in the present specification.

Second, however, the criticality of using the claimed fly-ash containing cenospheres as opposed to any other type of alumina/silica mixture may be readily appreciated by inspecting the much lower thermal conductivities afforded by the present invention as compared to those of DRI-LITE® 65A and 79AC - for comparable temperatures.

Notably, the thermal conductivities of DRI-LITE® 65A, and DRI-LITE® 79AC are 1.80 and 2.70, respectively at 800°F. At the comparable temperature of 732°F, "PYROFLOW" of the present invention has a thermal conductivity of 1.19. This is the same result for the Example in the present specification. Even at

the high temperature of 1,200°F, the thermal conductivities for DRI-LITE® 65A, DRI-LITE® 79AC and "PYROFLOW" are 2.00, 3.10 and 1.82, respectively.

Thus, the thermal conductivities of the subject material of present claim 1 are uniformly lower than those of the compositions of Doza et al.

Additionally, as noted above, and in more detail, during the Interview with Examiner Kastler, several samples were displayed for inspection.

They were:

- a) DRI-ITE 65A "as received" (100g);
- b) DRI-LITE 65A "heated at 500°C for 2 hrs" (100g);
- c) PYROFLOW "as received" (100g); and
- d) PYROFLOW "heated at 500°C for 2 hrs (200g).

The visual and tactile observations made from these samples were as follows:

First, both DRI-LITE 65A samples a) and b) appear and feel as a rough, grainy composition, and when agitated, move as a rough, grainy composition with no fluidity.

Second, in contrast, PYROFLOW sample c) appears and feels as a fluid, fine dust, and when agitated, exhibits a notable fluidity.

Third, PYROFLOW sample d), which was heated at 500°C for 2 hours, has the appearance of a finished, solid product in the shape of a wheel. This is because of the binder content, which binder sets at a temperature of lower than 400°F. In contrast, the DRI-LITE 65A sample b) remained as a rough, grainy powder even after being heated to 500°C for 2 hours. This latter difference is due to the incorporation of a binder in the PYROFLOW composition which has a setting temperature of less than 400°F.

By using either anhydrous boron oxide or boric acid, a low setting temperature is achieved for the present material, i.e., less than 400°F. Doza et al. fails to make any distinction between organic and inorganic binders. See column 4, lines 51-61. Further, this reference fails to either disclose or suggest the use of either boric acid or anhydrous boron oxide in order to achieve a low setting temperature.

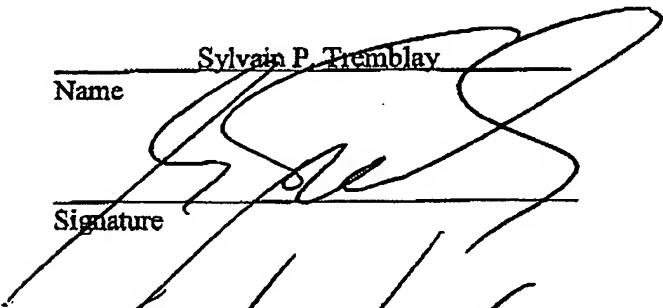
6) I am of the opinion that the presently claimed back-up insulating material affords properties, such as reduced thermal conductivity and reduced setting temperature, that would not have been expected by one skilled in the art at the time the present invention was made.

7) I am also of the opinion that the presently claimed back-up insulating material and the properties thereof are important and commercially significant.

8) I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine, or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

9) Further, declarant sayeth not.

Name Sylvain P. Tremblay

Signature 

Date 01/11/2006



## Bibliography

1. Y. Trudel, C. Ciloglu & S. Tremblay, « Selectives additives to improve machinability of ferrous P/M parts », article présenté au 1984 International P/M Conference and Exhibition tenu à Toronto en juin 1984.
2. S. Tremblay, pagne & R. Angers, « Fabrication of dense WC-Co spheroidal particles », article présenté au 23<sup>rd</sup> Annual Conference of Metallurgists tenu à Québec en août 1984.
3. S. Tremblay, B. Champagne & R. Angers, « Mechanical properties of copper-infiltrated, low-alloy P/M steel and stainless steel », Progress in Powder Metallurgy, Vol. 42, edited by E.A. Calson & G. Gaines, 1986.
4. S. Tremblay & R. Angers, « Spheroidization of WC-Co agglomerates », The International Journal of Powder Metallurgy, Vol. 25, No. 3, 1989, pp. 179-184.
5. S. Pelletier, R. Angers et S.P. Tremblay, « Caractéristiques mécaniques et microstructurales de composites Al2O3 », présenté au 57 ième congrés de l'ACFAS, Montréal, mai 1989.

6. R. Allem, M. Caron, G. L'espérance, I. DeSousa et S.P. Tremblay, Caractérisation morphologique, cristallographique et chimique de trichites de carbure de silicium », présenté au 57 ième congrès de l'ACFAS, Montréal, mai 1989.
7. M.K. Jain, S.P. Tremblay & D.J. Lloyd, « Evaluation of SiC platelets in Metal Matrix Composites », article présenté au 1990 Annual Meeting en février 90 à Anaheim, California.
8. S.P. Tremblay, M.K. Jain et D.J. Lloyd, « Evaluation des plaquettes de SiC dans les composites métalliques », présenté au 58ième congrès de l'ACFAS, Québec, 1990.
9. D. Baril et S.P. Tremblay, »SiC platelets reinforced Si<sub>3</sub>N<sub>4</sub> composites », article présenté au 93<sup>rd</sup> Ceramic Conference and Exposition à Cincinnati, Ohio, en mai 1991.
10. D. Baril, S.P. Tremblay and M. Fiset, « Silicon carbide platelet-reinforced silicon nitride composites », Journal of Materials Science, Vol. 28, November 1993, pp. 5486-5494.

11. S.P. Tremblay, R. Breault, J. Lachance and Y. Huard, « Market opportunities for plasma dross residues », article présenté au Light Metals 1995, Las Vegas, Février, 1995.
12. C. Brochu, R. Dault, J. Déry & S.P. Tremblay, "Liquid metal filtration and distribution using fiberglass fabric", Light Metal, 1996, 839-844.
13. S.P. Tremblay, "Filtering and distribution of molten aluminium using a fibreglass medium", Presented at the 98 Aluminium Asia Conference in Kuala Lumpur, Malaysia, 1998.
14. S.P. Tremblay & C. Brochu, "The use of fiberglass fabric to filter and distribute molten aluminium", Aluminium Times, April-May, Vol. 1, No. 2, 1999.
15. S.P. Tremblay & R. Green, "The manufacturing, Design and Use of Combo Bag Distributors in Sheet Ingot Casting", Continuous Casting Conference, Frankfurt, Germany, November 13-15<sup>th</sup>, 2000.
16. D. Weis and S.P. Tremblay, "Phosphate Bonded Wollastonite Refractories for Continuous Strip Casting of Aluminium", Aluminium World, Spring 2001.

17. S.P. Tremblay and M. Lapointe, "The manufacturing, design and use of a new reusable molten metal distributor for sheet ingot casting", Presented at TMS in February 2002.
18. S.P. Tremblay and M. Ruel, "The manufacturing, design and use of TF combo bag for sheet ingot casting", presented at TMS in March 2003.
19. S.P. Tremblay, A. Buchholz, M. Lapointe, "Reusable Molten Aluminum Distributor (ReMAD) for Sheet Ingot Casting - Mathematical simulation results Aluminium World", May 2003.
20. S.P. Tremblay, "The efficient TF combo bags for DC sheet ingot casting", présenté au X International Conference-Exhibition Aluminium of Siberia-2004, held in Krasnoyarsk, Russia on September 7-10, 2004.
21. S.P. Tremblay and M. Vincent, " Developement and use of a new composite material for aluminum contact applications", présenté au TMS 2005 a San Francisco, CA en février 2005.
22. D. Larouche and S.P. Tremblay, "TF combo bag - Mathematical simulation results and up-date", présenté au Pyrotek Conference, Dubai, UAE, November 2005.

# PRODUCT BULLETIN

Allied Mineral Products, Inc.  
2700 Scioto Parkway • Columbus, Ohio 43221 • 614/876-0244 • Fax 614/876-0981



## DRI-LITE™ 79AC

### General Information

Dri-Lite™ 79AC is an insulating dry-vibratable designed for use as a safety lining in aluminum industry melting and holding equipment. This product is well suited as an insulating backup lining for reverberatory furnaces, degassing chambers, metal transfer vessels, launders and precast shapes. Dri-Lite™ 79AC can also be used in similar applications processing copper alloys and other low temperature non-ferrous metals. This product offers the following benefits and features:

- > Aluminum resistance
- > Excellent insulating values
- > Completely dry with heat-set bond

### Technical Data

#### Chemical Analysis

Al <sub>2</sub> O <sub>3</sub>	42.9%
SiO <sub>2</sub>	40.6%
Fe <sub>2</sub> O <sub>3</sub>	1.3%
Alkali	1.2%

Material Required .....	82 lbs. per cubic foot (1.31 g/cm <sup>3</sup> )
Grain Size .....	3 mesh (6 mm) and finer
Maximum Use Temperature .....	2500°F (1370°C)
Installation Method .....	Ram, vibration
Storage Life .....	12 months

Packaged in 35-lb. (16 kg) multi-wall paper bags. Palletized 64 bags (2240 lbs. or 1015 kgs) per 42" x 42" pallet, protected with stretch wrap. Also available in bulk packaging. Storage beyond 12 months is not recommended. Store in a dry location to avoid moisture pickup.

Matrix Refractories, Inc. supplies a *complete line* of monolithic refractories for the metals industry. For more information or a complete evaluation of your refractory requirements, please contact your local Matrix representative.

**Warning:** Contains aluminum oxide, aluminum silicates, proprietary ingredients and silica. The International Agency for Research on Cancer (IARC) has classified crystalline silica inhaled in the form of quartz or cristobalite carcinogenic to humans. Refer to Material Safety Data Sheet for additional information and disposal instructions. Avoid breathing dust. Wear NIOSH approved respirator during installation, removal, and disposal of product to prevent inhalation of dust.

Issued 8/10/99  
©1999 Matrix Refractories, Inc.



THE INFORMATION STATED IS BASED ON THE BEST LABORATORY AND LITERATURE DATA AND IS NOT A WARRANTY OR A GUARANTEE, NOR A VIOLATION OF ANY PATENT. IT IS PRESENTED FOR CONSIDERATION AND VERIFICATION ONLY, AND IS NOT TO BE USED FOR SPECIFICATION PURPOSES.



## LABORATORY TEST BAR DATA DRI-LITE™ 79AC

### Density, pcf (g/cm<sup>3</sup>)

After firing to:

1000°F ( 540°C)	83 (1.33)
1500°F ( 815°C)	84 (1.35)
2000°F (1095°C)	84 (1.35)

### Permanent Linear Change, %

After firing to:

1000°F ( 540°C)	-0-
1500°F ( 815°C)	-0.1
2000°F (1095°C)	-0.4

### Modulus Of Rupture, psi (MPa)

After firing to:

1000°F ( 540°C)	25 (0.2)
1500°F ( 815°C)	205 (1.4)
2000°F (1095°C)	285 (2.0)

### Thermal Conductivity, BTU-in/°F-hr-ft<sup>2</sup> (W/mK)

By Hot-Wire Method (ASTM C-1113)

At:

400°F ( 205°C)	2.20 (0.32)
800°F ( 425°C)	2.70 (0.39)
1200°F ( 650°C)	3.10 (0.45)
1600°F ( 870°C)	3.45 (0.50)
2000°F (1093°C)	3.90 (0.56)
2200°F (1204°C)	4.02 (0.58)



Allied Mineral Products, Inc.  
2700 Scioto Parkway  
Columbus, Ohio 43221  
614/876-0244  
Fax 614/876-0981  
E-mail: [matrix@alliedmin.com](mailto:matrix@alliedmin.com)  
[www.alliedmatrix.com](http://www.alliedmatrix.com)

# PRODUCT BULLETIN

Allied Mineral Products, Inc.  
2700 Scioto Parkway • Columbus, Ohio 43221 • 614/876-0244 • Fax 614/876-0981



## DRI-LITE™ 65A

### General Information

DRI-LITE™ 65A is an *aluminum-silicate* based insulating dry-vibratable. This product exhibits very low density as well as excellent insulating properties. DRI-LITE™ 65A can also be used in service at temperatures as high as 2550°F. It has been designed for insulating backup in anode carbon baking furnaces, aluminum launder precast shapes, as well as many other applications where minimizing heat loss is crucial. This product offers the following benefits and features:

- > Completely dry with heat set bond
- > Lightweight, but high service temperature
- > Low thermal conductivity

### Technical Data

#### Chemical Analysis

Al <sub>2</sub> O <sub>3</sub>	37.8%
SiO <sub>2</sub>	59.0%
TiO <sub>2</sub>	1.2%
CaO	0.6%
Fe <sub>2</sub> O <sub>3</sub>	0.6%
Others	0.8%

Material Required	60 lbs. per cubic foot (0.96 g/cm <sup>3</sup> )
Grain Size	4 mesh (5 mm) and finer
Maximum Use Temperature	2550°F (1400°C)
Installation Method	Fork and dry ram
Storage Life	12 months

### Typical Physical Properties:

#### Permanent Linear Change (%)

After heating to:

1400°F (760°C)	0.3
1800°F (980°C)	0.1
2400°F (1315°C)	0.1

#### Thermal Conductivity, BTU-in/°F-hr-ft<sup>2</sup> (W/mK)

By Hot-Wire Method (ASTM C-1113)

At:

400°F (205°C)	1.50 (0.22)
800°F (425°C)	1.80 (0.26)
1200°F (650°C)	2.00 (0.29)
1500°F (816°C)	2.20 (0.32)
1800°F (982°C)	3.18 (0.46)

Packaged in 35-lb. (16 kg) multi-wall paper bags. Palletized 64 bags (2240 lbs. or 1015 kgs) per 42" x 42" pallet, protected with stretch wrap. Also available in bulk packaging. Store in a dry location to avoid moisture pickup.

MATRIX REFRACTORIES DIVISION supplies a *complete line* of monolithic refractories for the aluminum industry. For more information or a complete evaluation of your refractory requirements, please contact your local MATRIX representative.

**Warning:** Contains aluminum oxide, aluminum silicate, and silica. The International Agency for Research on Cancer (IARC) has classified crystalline silica inhaled in the form of quartz or cristobalite carcinogenic to humans. Refer to Material Safety Data Sheet for additional information and disposal instructions. Wear NIOSH approved respirator during installation, removal, and disposal of product to prevent inhalation of dust. In case of eye contact, flush immediately and repeatedly with water and consult a physician. For safest use and optimum performance, proper practices must be followed.

Issued 7/22/99  
©1998 MATRIX REFRACTORIES DIVISION  
Allied Mineral Products, Inc.



THE INFORMATION STATED IS BASED ON THE BEST LABORATORY AND LITERATURE DATA AND IS NOT A WARRANTY OR A GUARANTEE, NOR A VIOLATION OF ANY PATENT. IT IS PRESENTED FOR CONSIDERATION AND VERIFICATION ONLY, AND IS NOT TO BE USED FOR SPECIFICATION PURPOSES.



PRESIDENTIAL AWARD FOR EXPORTS

# PYROFLOW (L&D)

## FREE-FLOWING DRY INSULATING MATERIAL

PYROFLOW (L & D) is a free-flowing dry insulating material. The product flows like water, exhibits very low density as well as excellent insulating properties. PYROFLOW (L & D) can be used in service temperature up to 900°C (1650°F). It has been designed as insulating backup for working linings in the aluminum industry for minimizing heat loss.

### BENEFITS:

- water free
- no organic binder
- free-flowing
- light weight
- low thermal conductivity
- heat setting bond developed as low as 200°C (392°F)
- non-wetting to molten aluminium

### TECHNICAL DATA:

CHEMICAL ANALYSIS (%) WEIGHT BASIS	
Al <sub>2</sub> O <sub>3</sub>	23 – 28
SiO <sub>2</sub>	51 – 61
Crystalline Silica	0.5 – 0.6
TiO <sub>2</sub>	0.8 – 0.9
Fe <sub>2</sub> O <sub>3</sub>	3.0 – 4.0
B <sub>2</sub> O <sub>3</sub>	5 – 15
CaF <sub>2</sub>	0 – 5
Dust suppressant	0 – 0.5
Density	25 – 30 (lb/ft <sup>3</sup> )
Maximum use temperature	900°C (1650°F)
Installation method	free-flowing
Storage life	24 months

### THERMAL CONDUCTIVITY:

MEAN TEMPERATURE		$\frac{\text{BTU} \cdot \text{in}}{\text{ft}^2 \cdot \text{hr} \cdot ^\circ\text{F}}$	$\frac{\text{W}}{\text{m} \cdot ^\circ\text{K}}$
°F	°C		
275	135	0.85	0.122
504	262	1.01	0.145
732	389	1.19	0.172
961	516	1.42	0.205
1202	650	1.82	0.262

*Patent Pending*

Advanced Metals Processing Technology

# Pyrotek

CORPORATE OFFICE • EAST 9503 MONTGOMERY AVENUE • SPOKANE, WA 99206  
PHONE (509) 926-6212 • FAX (509) 927-2408 • PY-05-04-761  
[www.pyrotek.info](http://www.pyrotek.info) • e-mail: [info@pyrotek.info](mailto:info@pyrotek.info)



**This Page is Inserted by IFW Indexing and Scanning  
Operations and is not part of the Official Record**

**BEST AVAILABLE IMAGES**

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images include but are not limited to the items checked:

- ☒ **BLACK BORDERS**
- ☐ **IMAGE CUT OFF AT TOP, BOTTOM OR SIDES**
- ☐ **FADED TEXT OR DRAWING**
- ☐ **BLURRED OR ILLEGIBLE TEXT OR DRAWING**
- ☐ **SKEWED/SLANTED IMAGES**
- ☐ **COLOR OR BLACK AND WHITE PHOTOGRAPHS**
- ☐ **GRAY SCALE DOCUMENTS**
- ☐ **LINES OR MARKS ON ORIGINAL DOCUMENT**
- ☐ **REFERENCE(S) OR EXHIBIT(S) SUBMITTED ARE POOR QUALITY**
- ☐ **OTHER:** \_\_\_\_\_

**IMAGES ARE BEST AVAILABLE COPY.**

**As rescanning these documents will not correct the image problems checked, please do not report these problems to the IFW Image Problem Mailbox.**